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IS 5254 (1980): Acetanilide, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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“Knowledge is such a treasure which cannot be stolen”

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IS : 5254 - 1980

Indian Standard REAFFIRMED
SPECIFICATION FOR
ACETANILIDE, TECHNICAL
(*First Revision*)

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

SPECIFICATION FOR ACETANILIDE, TECHNICAL

(First Revision)

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Indian Standard
SPECIFICATION FOR
ACETANILIDE, TECHNICAL
(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 10 August 1980, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1969. As a result of the periodical review of the quality of product available and that specified in the Indian Standard as well as various overseas standards, it was decided to take up this revision.

0.2.1 In the present revision the requirement for assay has been modified. The bromination method for the determination of acetanilide content has been replaced by the diazotization method and the crystallizing point characteristic has been substituted for the melting point.

0.3 Acetanilide (N-phenylacetamide) is used as an intermediate for basic drugs and dyestuffs. It is also used as rubber accelerator and in the manufacture of cellulose ester varnishes. Its use as an analgesic is banned because its habitual use for relief of headache has resulted in damage to blood forming organs followed by anaemia and cyanosis.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for acetanilide suitable for industrial purpose (*see 0.3*).

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be a white to buff coloured crystalline powder or white to grey-brown flakes. It shall be soluble in hot water and in common organic solvents.

2.2 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of the appendix is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR ACETANILIDE
(Clauses 2.2, B-6.1 and B-6.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHODS OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, <i>Min</i>	99.0	A-2
ii)	Crystallizing point, <i>Min</i>	112.5°C	A-3
iii)	Free acetic acid, percent by mass, <i>Max</i>	0.5	A-4
iv)	Free aniline, percent by mass, <i>Max</i>	0.2	A-4
v)	Moisture, percent by mass, <i>Max</i>	0.2	A-5
vi)	Sulphated ash, percent by mass, <i>Max</i>	0.2	A-6

3. PRECAUTIONS IN HANDLING

3.1 Acute poisoning may occur if several grams of acetanilide is ingested. It may also cause contact dermatitis, and inhalation or ingestion may cause eruption on the skin. Hence, care should be exercised while handling acetanilide to prevent ingestion even by inhalation.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in cardboard boxes, bottles, fibre drums, multi-walled paper sacks or as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 The containers shall be marked with the following cautionary note:

‘ DO NOT INGEST OR INHALE ’

4.2.2 The containers shall also be marked with the following information:

- a) Manufacturer's name and his recognized trade mark, if any;
- b) Name of the material;
- c) Mass of the material in the container; and
- d) Batch number.

4.2.2.1 The material may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative samples of the material shall be drawn and their conformity determined as prescribed in Appendix B.

APPENDIX A

(Clause 2.2, and Table 1)

METHODS OF TEST FOR ACETANILIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF ACETANILIDE CONTENT

A-2.0 Outline of the Method — The material is hydrolysed with hydrochloric acid. Aniline formed is diazotized with sodium nitrite solution, excess of which is back titrated with standard *para*-nitroaniline solution using potassium starch iodide test paper as indicator.

A-2.1 Reagents

A-2.1.1 Concentrated Hydrochloric Acid — see IS : 265-1976†.

*Specification for water for general laboratory use (*second revision*).

†Specification for hydrochloric acid (*second revision*).

A-2.1.2 Standard Sodium Nitrite Solution — 0.2 N.

A-2.1.3 Standard Para-nitroaniline Solution — 0.2 N.

A-2.1.4 Potassium Starch Iodide Test Paper

A-2.1.5 Potassium Bromide — Crystals.

A-2.2 Procedure — Avoiding exposure to air, weigh accurately about 5 g of the sample previously ground to a powder and transfer quantitatively to a 500 ml round-bottomed flask. Add 200 ml water and 50 ml hydrochloric acid. Attach a reflux condenser to the round-bottomed flask and heat under reflux for 30 minutes. Cool to room temperature. Transfer the reaction mixture quantitatively to a 500-ml volumetric flask and dilute with water to the mark.

A-2.2.1 Pipette out 100 ml aliquot from the volumetric solution prepared as above into a one-litre beaker containing 300 ml water. Add 10 g of potassium bromide and cool to 20°C.

While stirring mechanically, titrate, dropwise, at this temperature with standard sodium nitrite solution which is added below the surface of the solution through a thistle funnel. Add the nitrite solution as rapidly as it is consumed. Test the solution by spotting on potassium starch iodide test paper. There should be no blue colouration on the test paper. Before 2 ml of the expected end point, the addition of nitrite solution is continued in 0.5 ml lots until a small excess has been added. A small excess is shown when a drop of the titrated mixture on potassium iodide starch test paper produces an immediate medium strong blue colouration which persists for 2 minutes. After 2 minutes, back titrate the excess nitrite with standard para-nitroaniline solution till the blue colouration appearing on spotting the titrated mixture on potassium iodide starch test paper, just disappears.

A-2.3 Calculation

1 ml of 1 N NaNO_2 = 0.1352 g acetanilide (M. wt. 135.2)

$$\text{Acetanilide content, percent by mass} = \frac{(V_1 N_1 - V_2 N_2) \times 6760}{A \times M}$$

where

V_1 = volume in ml of standard sodium nitrite solution added,

N_1 = normality of sodium nitrite solution,

V_2 = volume in ml of standard para-nitroaniline solution used in the back titration,

N_2 = normality of para-nitroaniline solution,

A = aliquot taken for titration, and

M = mass in g of the sample taken.

NOTE — Correction shall be made for free aniline content. Deduct 1.45 percent acetanilide for every 1 percent aniline from the value obtained in A-2.3 above.

A-3. DETERMINATION OF CRYSTALLIZING POINT

A-3.1 Procedure — Carry out the determination of crystallizing point according to the method as prescribed in IS : 5813-1970*.

A-4. DETERMINATION OF FREE ACETIC ACID AND FREE ANILINE

A-4.1 Reagents

A-4.1.1 Standard Sodium Hydroxide Solution — 0.1 N.

A-4.1.2 Phenolphthalein Indicator — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

A-4.1.3 Potassium Bromide — crystals.

A-4.1.4 Hydrochloric Acid — see IS : 265-1976†.

A-4.1.5 Standard Sodium Nitrite Solution — 0.1 N. Standardize against sulphanilic acid as follows:

Dissolve an accurately weighed quantity of sulphanilic acid (about 0.4 g) dried by heating at 100°C to constant mass, in 50 ml of water and 5 ml of hydrochloric acid in a beaker. Cool to 15°C, add about 30 g of crushed ice, then titrate slowly with sodium nitrite solution stirring vigorously until pale-blue or greyish-blue colour is produced immediately when a glass rod dipped in the titrated solution is spotted on starch-iodide paper. When the titration is complete, the end point should be reproducible after the solution has been standing for a minute. Calculate the normality of the sodium nitrite solution as given below:

1 g of sulphanilic acid = 0.398 4 g of sodium nitrite.

A-4.1.6 Starch-Iodide Paper — Take 0.5 g of starch in 10 ml of cold water and slowly pour it with constant stirring into 100 ml of boiling water. Boil it gently until the mixture is thin and translucent. Allow it to cool. Dissolve 0.5 g potassium iodide and 1 g of zinc chloride in it. Dip the filter papers in the above solution and dry them. Store in dark.

A-4.2 Prepared Solution — Weigh accurately about 50 g of the material and transfer in a 2-litre flask using 1 000 ml water. Attach a condenser to the flask and heat to dissolve completely. Recrystallize the material by cooling in an ice bath. Filter and make up the filtrate to 1 000 ml with water in a 1 000-ml one mark graduated flask.

*Method for the determination of crystallizing point.

†Specification for hydrochloric acid (second revision).

A-4.3 Procedure

A-4.3.1 Take 500 ml of the prepared solution (*see A-4.2*) in a 1 000-ml beaker and titrate with standard sodium hydroxide solution using phenolphthalein indicator.

A-4.3.2 Cool the titrated solution (*see A-4.3.1*) to 0°C by adjusting the beaker in an ice bath and add 5 g of potassium bromide and 50 ml of hydrochloric acid. Titrate, while stirring mechanically, at 0°C with standard sodium nitrite solution using starch-iodide paper. Note the end point when an immediate pale-blue ring appears on starch-iodide paper, which can be obtained repeatedly for 2 minutes without further addition of nitrite solution.

A-4.4 Calculation

$$\text{a) Free acetic acid, percent by mass} = \frac{12 V_1 N_1}{M}$$

where

V_1 = volume in ml of standard sodium hydroxide solution used up,

N_1 = normality of standard sodium hydroxide solution, and

M = mass in gram of the material taken for the test (*see A-4.2*).

$$\text{b) Free aniline, percent by mass} = \frac{18.6 V_2 N_2}{M}$$

where

V_2 = volume in ml of standard sodium nitrite solution used up,

N_2 = normality of standard sodium nitrite solution, and

M = mass in g of the material taken for the test (*see A-4.2*).

A-5. DETERMINATION OF MOISTURE

A-5.0 General — The moisture is determined by the Karl Fischer method.

A-5.1 Procedure — Weigh accurately 1 g of the material and determine the moisture content by the procedure given in IS : 2362-1973*.

*Determination of water by Karl Fischer method (*first revision*).

A-6. DETERMINATION OF SULPHATED ASH

A-6.0 Outline of the Method — The material is ignited and burnt until only ash and carbon remain. After cooling, the charred residue is treated with sulphuric acid and heated until oxidation of the carbon is practically complete. The residue is then cooled and weighed.

A-6.1 Apparatus

A-6.1.1 Crucible — squat form; of silica, porcelain or platinum.

A-6.1.2 Bunsen Burner and Muffle Furnace

A-6.2 Reagents

A-6.2.1 Concentrated Sulphuric Acid — relative density 1.84 (see IS : 266-1977*).

A-6.2.2 Concentrated Nitric Acid — relative density 1.40 (see IS : 264-1976†).

A-6.3 Procedure — Weigh accurately about 1 to 2 g of the material in the tared crucible and ignite until thoroughly charred. Cool, moisten the residue with 1 ml of concentrated nitric acid and 1 ml of concentrated sulphuric acid and cautiously ignite until the carbon is completely consumed. Continue the ignition in a place protected from air currents and using as low a temperature as possible to effect the combustion of the carbon. When the carbon has completely disappeared, cool the crucible in a desiccator and weigh. Repeat the ignition for an additional period of 15 minutes, cool and weigh. Two successive weighings shall not differ more than 0.5 mg.

A-6.4 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{100 (M_1 - M_2)}{M}$$

where

M_1 = mass in g of the ignited residue with the crucible,

M_2 = mass in g of the crucible, and

M = mass in g of the material taken for the test.

A P P E N D I X B

(Clause 5.1)

SAMPLING OF ACETANILIDE**B-1. GENERAL REQUIREMENTS OF SAMPLING**

B-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

*Specification for sulphuric acid (second revision).

†Specification for nitric acid (second revision).

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination (*see also 3.1*).

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry and air-tight glass containers or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, batch number and other important particulars of the consignment.

B-1.8 Samples shall be stored in a cool and dry place.

B-2. SAMPLING INSTRUMENT

B-2.1 The sampling instrument is a closed type sampling tube, undivided (*see Fig. 1*). It consists of two concentric cylindrical tubes made of a metal which is not affected by the material being sampled (preferably of stainless steel), one closely fitting into the other throughout their length so that it is possible to rotate one tube within the other, a suitable handle being provided for the purpose. Longitudinal openings of about one-third the circumference are cut in both tubes throughout their length. In one position the two openings coincide and admit the material into the hollow inner tube. By rotating the inner tube through 180° the opening is tightly closed and a 'core' of material being enclosed therein may be withdrawn. This type of sampler is usually provided with a locking arrangement so that the tubes are held together in any desired position. The outer tube is provided with a sharp conical end to facilitate penetration but the base of the cone shall be closed so that no material is entrapped in this portion. The height of the cone shall be equal to its base diameter. The whole instrument shall be of sufficient length to penetrate an entire diagonal of the container being sampled. The diameter of the inner cylindrical space may vary from 20 to 40 mm, proportionately to the length. A length of 150 cm and a diameter of 30 mm can cater for most needs.

B-2.1.1 *Use of Sampling Instrument* — The instrument is inserted in closed position in an oblique direction till it touches bottom. The material is admitted by rotating and opening the tubes and finally closing them, withdrawing the sample in this process. In case the minimum quantity of material required for test from each container is more than the capacity of the instrument, further 'cores' shall be taken from different

parts of the same container, such that they are at least 75 mm in the case of drums, bags, etc, and 25 mm in the case of small containers, from the wall of the container. In all cases the instrument shall be inserted till it touches bottom so that an entire cross section is withdrawn.

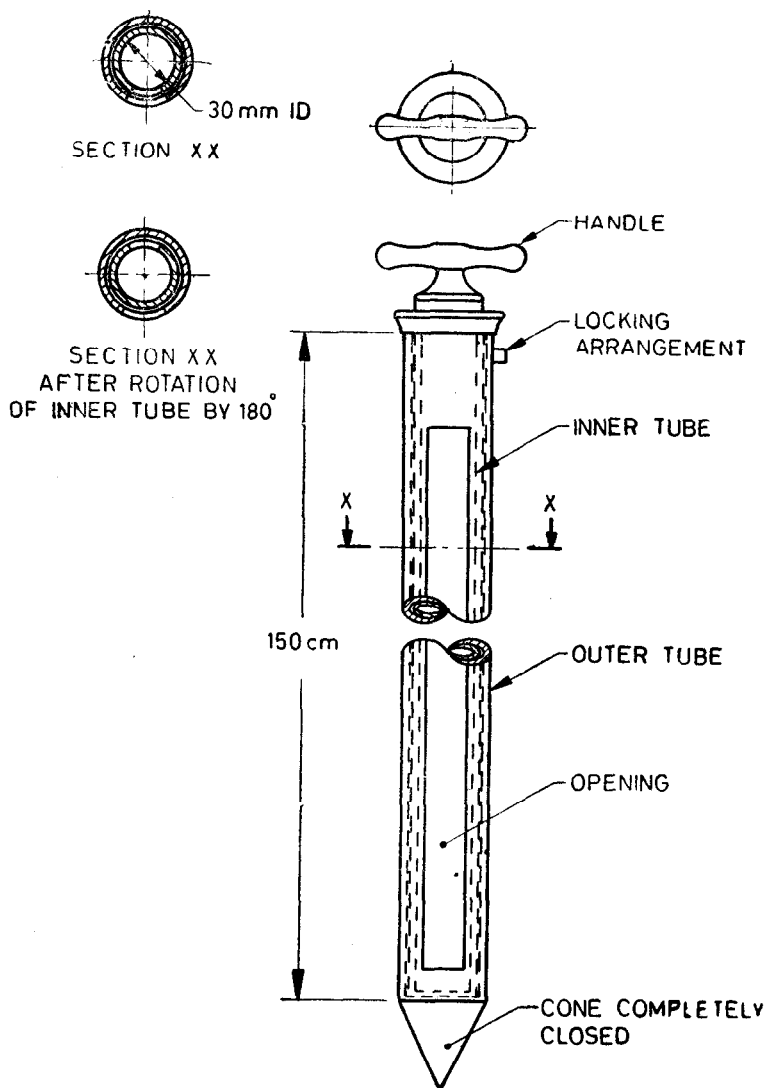


FIG. 1 CLOSED TYPE SAMPLING TUBE, UNDIVIDED

B-3. SCALE OF SAMPLING

B-3.1 Lot — All the containers in a single consignment of the material drawn from single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, the containers belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

B-3.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for drawing the samples shall depend upon the size of the lot and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FROM LOTS OF DIFFERENT SIZES

LOT SIZE (N)	NO. OF CONTAINERS TO BE SELECTED (n)
4 to 25	3
26 „ 50	4
51 „ 100	5
101 „ 150	6
151 „ 300	7
301 and above	8

NOTE — When the size of the lot is three or less, all the containers shall be sampled.

B-3.3 These containers shall be selected at random from the lot and in order to ensure the randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them in one order as 1, 2, 3, up to r , and so on, where r is the integral part of N/n (N being the lot size and n the number of containers to be selected). Every r th container thus counted shall be withdrawn to give sample for test.

B-4. TEST SAMPLE AND REFEREE SAMPLE

B-4.1 From each of the containers selected as in **B-3.2**, draw with the sampling instrument of an appropriate size small portions of the material from different parts of the container. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes.

B-4.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

B-4.3 From the samples (*see* B-4.2) representing different containers selected in B-3.2, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample sufficient to carry out testing for the characteristics specified. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-4.4 The remaining portion of the material in the samples (*see* B-4.2) from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the n containers selected shall be for the purchaser, another for the supplier and the third for the referee.

B-4.5 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in B-1.7.

B-4.6 The referee samples consisting of a composite sample and a set of n individual samples, shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

B-5. TESTS

B-5.1 Tests for description (*see* 2.1), determination of acetanilide content and crystallizing point shall be conducted on each of the individual sample.

B-5.2 Tests for the determination of free acetic acid, free aniline, moisture and sulphated ash shall be conducted on the composite sample.

B-6. CRITERIA FOR CONFORMITY

B-6.1 For Individual Samples — The lot shall be declared as conforming to the requirements of description, acetanilide content and crystallizing point, if each of the test results satisfies the corresponding requirement given in 2.1 and Table 1.

B-6.2 For Composite Sample — For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample (*see* B-4.2), the test results shall satisfy the relevant requirements given in Table 1.

(Continued from page 2)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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22E Kalpana Area	BHUBANESHWAR 751014	5 36 27
Ahimsa Bldg, SCO 82-83, Sector 17C	CHANDIGARH 160017	2 83 20
5-8-56C L. N. Gupta Marg	HYDERABAD 500001	22 10 83
R 14 Yudhister Marg, C Scheme	JAIPUR 302005	6 98 32
117/418 B Sarvodaya Nagar	KANPUR 208005	8 12 72
Patliputra Industrial Estate	PATNA 800013	6 28 08
Hantex Bldg (2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27